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High Temperature Composites

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HIGH TEMPERATURE COMPOSITES

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The purpose of this paper is to review the current state of development of new composite materials for advanced aircraft engines. The advantages and disadvantages of Ti-base, NiAl-base, and $MoSi_2$-base composites as replacements for today's Ni-base superalloys are discussed from the standpoint of key technical issues, current status, and future directions. Results describing progress in both improved understanding of the mechanisms of deformation and fracture, and improved material performance will be covered.

KEY WORDS *composites, Ti alloys, Ti-aluminides, NiAl, $MoSi_2$-base, jet engines*

1. Introduction

Advanced composites are a key to the development of the next generation of civil transport aircraft engines. The driving forces for the development of advanced engines include both mission-enabling capabilities and reduced life-cycle costs. An example of a mission-enabling capability is an advanced, environmentally friendly engine for a future supersonic civil transport. Composites and other advanced materials will play a key role in meeting the requirements for this engine, which include very low NO_x emissions, stringent noise regulations, low engine weight and acceptable engine performance to make such an engine economically attractive(1). Numerous applications for composites are also envisioned in advanced subsonic aircraft, such as the high bypass ratio turbofans currently being developed(2). High temperature composites research at NASA Lewis Research Center is primarily focused on aircraft engines. The effort in metallic systems includes both metal- and intermetallic matrix composites. The materials are targeted for a wide range of engine applications from the fan to the exhaust nozzle, which in turn corresponds to a range in service temperatures from 300 to 1100°C. This paper will focus on those composites that have shown significant progress and have matured to a point where further development appears justified. Technical highlights regarding both improved material performance plus improved understanding of the controlling mechanisms will be included. In particular, Ti-base composites, with both Ti-alloys and Ti-aluminides as matrices, are attractive for low and intermediate temperature applications. NiAl based composites are very attractive at higher temperatures, and finally $MoSi_2$ based composites have shown promise at even higher temperatures.

In order to be of use in jet engines, materials require a balance of properties, and Fig. 1 depicts six of the most important properties, namely toughness, processability, thermal conductivity, oxidation resistance, temperature capability, and specific strength or life. In a generalized view, Ni-base superalloys do possess the required balance, and their properties are plotted as a circle, indicating approximately equal achievement of all requirements. Because of this, they are used extensively today and they remain as the top competition for any advanced engine material. Fig. 1 also shows how intermetallics and composites compete with each other and with superalloys. $MoSi_2$ is the most ceramic-like of the intermetallics, with the highest temperature capability, good specific strength, and the lowest toughness and processability. Its conductivity is similar to superalloys, although somewhat lower in composite form. Its oxidation resistance is also quite good, including some recent results in solving the pest problem. NiAl is also a prime candidate, both in single crystal and composite form. It has the potential for high temperature capability and strength, and is tougher than silicides. NiAl can be processed relatively easily, has very high thermal conductivity, and has excellent oxidation resistance. Finally, Ti-based materials, including both aluminides and alloys, tend to have very good specific strength because of their low density, but do not have high temperature capability, primarily due to oxidation related problems. Process development of Ti-base materials has been progressing rapidly, and their toughness values have also become quite respectable.

So with this overview, a few important conclusions emerge. First, superalloys with their balance of properties have been and will continue to be difficult to surpass. This implies that trade-offs will be necessary: in order to take advantage of some benefits, some adjustments to handle the shortcomings must be made in either design or processing. Second, each of the material types have their niche, and each material will be favored in certain parts of an engine, or in different types of engines.

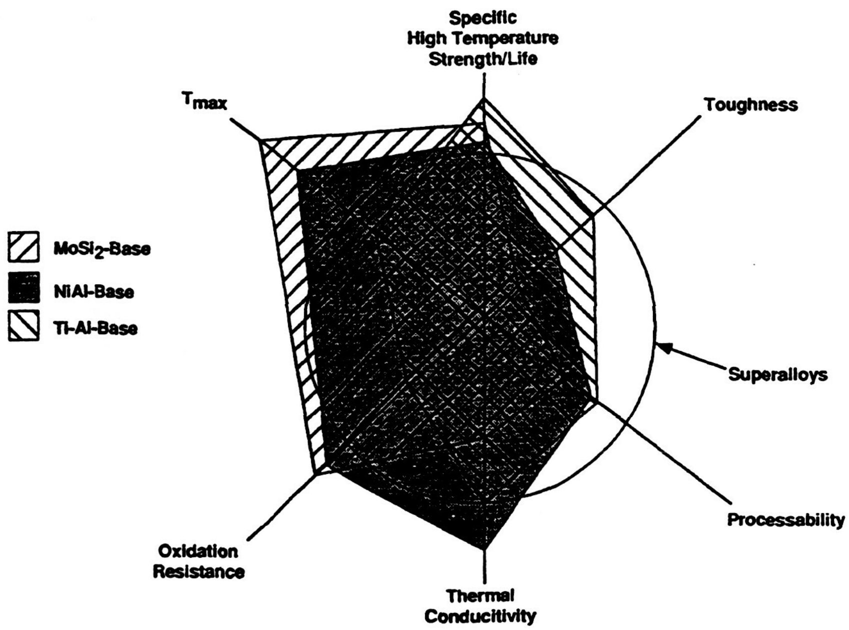
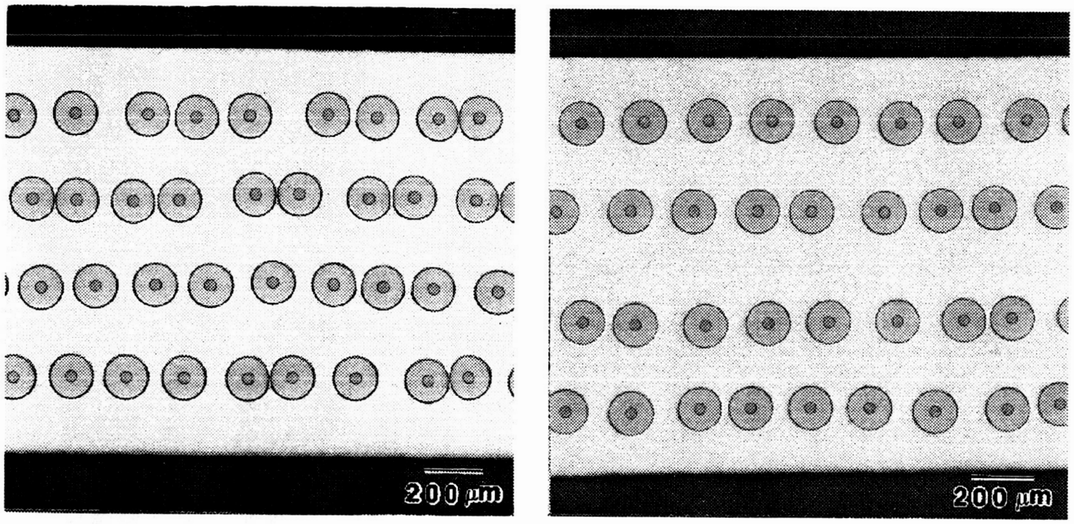


Figure 1. The balance of properties required for turbine engine application are represented by six axes. Generalizations of the properties of metallic and intermetallic composites are compared to superalloys.

2. Titanium Matrix Composites

Ti matrix composites have reached a stage of maturity that has included numerous successful engine tests of components in the fan and compressor that reach temperatures up to about 600°C. These composites are all made with continuous SiC fibers, and a variety of matrices. Ti alloys are the most mature and include current commercial alloys such as Ti-6Al-4V, Ti-15V-3Cr-3Al-3Sn, and others. A further advance in temperature capability can be achieved by going to an aluminide matrix, and Ti_3Al base α_2 alloys have been extensively studied(3), with a recent emphasis on alloys containing a high percentage of the orthorhombic phase(4). Again, these materials are best suited for low and intermediate temperature applications, primarily in the fan and compressor.

Several processing methods have been used to fabricate these composites, all with high degrees of success in terms of achieving good properties. Powder metallurgy methods require the use of binders in making the green composite, which then must be burned out before consolidation. For the powder cloth process, the green state consists of layers of powders held together with a binder which is alternated with fiber layers. When the binder is removed and consolidation is performed, there is movement of the powders and fibers, and some loss of the fiber spacing results, as shown in Fig. 2a. Touching fibers are of some concern(5), but in general properties of composites made from powder indicate that mechanical properties are competitive with other methods(3,6). Binder burn-out is not particularly difficult, and comparable interstitial impurity contents have been obtained in all these methods(3,6). Alternate stacking of matrix foils and fiber layers is also common, but foil making can be difficult and expensive. Thermal spray deposition has also been used extensively, along with vapor deposition such as sputtering or electron beam evaporation(7). These methods tend to have better fiber placement, as evidenced in Fig. 2b.



(a) (b)

Figure 2. Typical Ti matrix composite microstructures fabricated by: (a) powder cloth processing , and (b) spray deposition.

The basic tensile behavior of these types of composites is illustrated in Fig. 3. At 425°C, the stand alone Ti-15-3 matrix is reasonably strong at 600 MPa and ductile with about 18% strain to failure. The composite, when tested parallel to the fibers, is over twice as strong, twice as stiff, and is lower in density also. The specific stiffness alone is enough to make this material attractive for several components. However, the ductility is controlled by the SiC fiber, which has only about 1% total strain to failure. The advantages in the longitudinal properties are coupled with disadvantages in transverse properties, which are lower than the stand alone matrix(8). A wide body of evidence detailing the sequence of events during tensile deformation is summarized in Figure 4. Initially, the fiber and matrix both deform elastically in Stage I, until the onset of matrix yielding defines Stage II. The weakest fibers in the composite begin to fail at strains around 0.4 to 0.6 %, and these are usually distributed randomly throughout the gage. These fiber fractures accumulate until final failure occurs with little fiber pull-out at about 1% strain. The micrograph (Fig. 4b) shows that the fiber cracking present after tensile testing is periodic in nature and extends far away from the primary fracture surface. This again indicates that damage can accumulate gradually before the specimen fails(3,9,10).

It is clear that the fiber dominates longitudinal properties, and one must make sure that the processing method does not degrade the fibers. In general, studies on powder(3,6,9,10), foil(11), and thermal spray(6) methods all show that fiber strength degradation of 5 to 25% are possible, depending on the fabrication method and the specific fiber spool. It appears from comparison of plates made by different methods(6) and from specimens with intentionally mixed fiber strengths(9), that some fraction (near 10%) of lower strength fibers is tolerable.

Even though there are applications where a tradeoff in poor transverse properties can be tolerated in order to utilize the very high longitudinal properties, there is still a strong desire to understand and improve transverse properties. The mechanism for the low transverse strength is reasonably well understood. Based on analytical modeling(12), measurements of interfacial bond strengths(13), interrupted tensile tests(3), and acoustic emission testing(3) all point to an early onset of interfacial debonding under

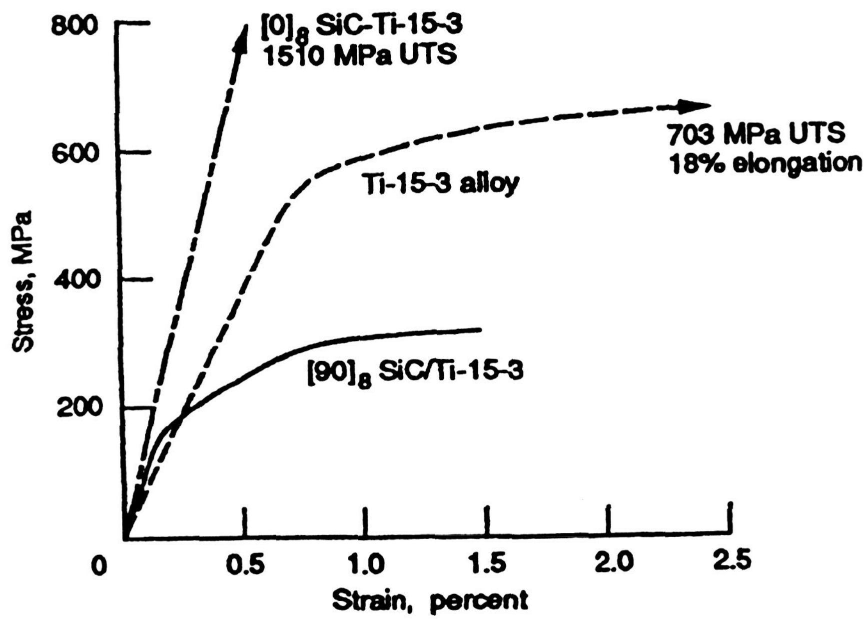


Figure 3. Typical tensile behavior of Ti matrix composites, as exemplified by SiC/Ti-15-3 tested at 425°C

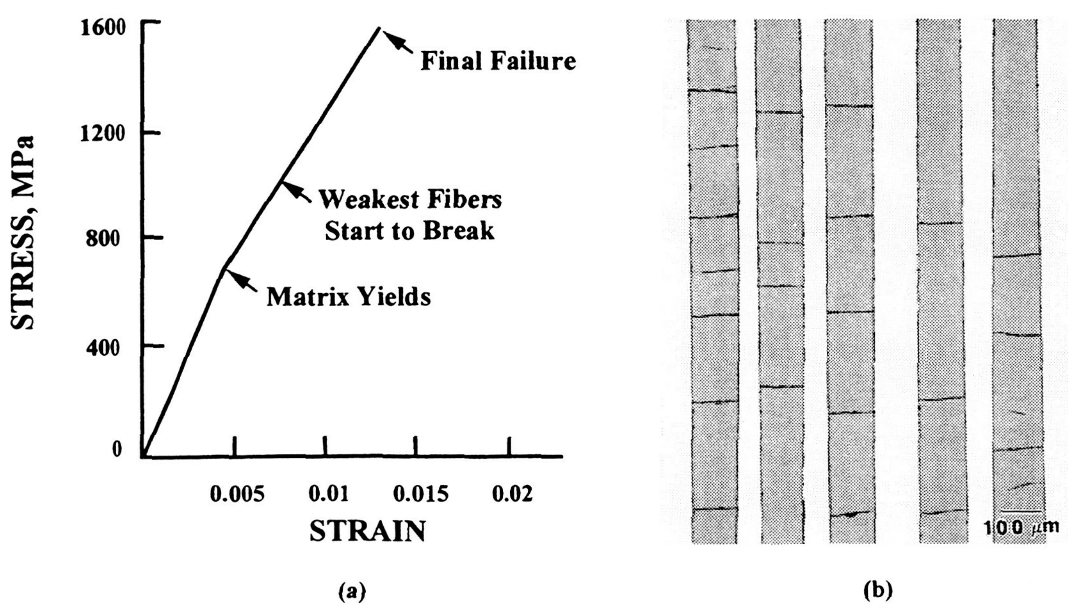


Figure 4. (a) general sequence of events during tensile deformation of Ti matrix composites. (b) Longitudinal section of SiC/Ti-24-11, showing fiber cracking after tensile testing.

tensile loading. At present there are no known methods for modifying the interface to improve the transverse properties without degrading the longitudinal properties. Thus, efforts have focused on matrix and process development to improve transverse properties(3,4). It has been shown(14) that for both Ti - 24Al - 11Nb and the orthorhombic matrices Ti - 20Al - 24Nb and Ti - 22Al - 23Nb, that both monolithic (matrix only) material and composites made by powder are stronger than the corresponding materials made from foil. Second, although it is still the case that the transverse properties of the composite are lower than its respective matrix, it is still of value to improve matrix strength in order to improve the composite's balance of properties. The orthorhombic alloys are much stronger than Ti - 24Al - 11Nb, and the transverse composite strength is about twice as high as the best composites of a few years ago.

Environmental resistance has surfaced as the other major technical barrier to the application of Ti composites. Although the oxidation resistance of Ti alloys in terms of oxide scale growth is not very good in comparison to Ni alloys, Ti-base alloys can still have reasonable oxidation rates up to 600°C and aluminides to 800°C(15). However, at temperatures much below those limits, fatigue-environment interactions which stem from oxygen embrittlement can prevent the large strength potential of the fibers from being reached. For SiC/ Ti-15-3 composites tested longitudinally in fatigue, life is reduced by a factor of 4 at 300°C when tested in air *versus* vacuum(16). Thermomechanical fatigue, where both temperature and stress are cycled simultaneously, is an even more severe test for composites, as a result of the residual stresses which can develop from the large differences in thermal expansion coefficients between the fiber and matrix. Although the non-isothermal tests did reduce life, it was found(17) that the environmental contribution to the reduced life was at least as important than the thermal expansion mismatch contribution. In a similar study(18) of α_2 composites, fatigue life at 425°C in air was about an order of magnitude shorter than in vacuum. This work also showed that the useful temperature limit of the composite is 400°C lower in air *vs* inert environments. Comparison of the data between the Ti alloy(16) and the Ti aluminide(18) also provides an indication of the matrix contribution to the fatigue life of composites, as shown in Fig. 5. Although the α_2 alloy has considerably more fatigue capability in vacuum, the actual life of the aluminide composite in air was only marginally better than that of the Ti-15-3 composite. Thus, these large life degradations caused by environmental effects also tend to reduce the beneficial effects of a stronger matrix. Again, this is not an oxidation rate effect, but an embrittlement of the matrix by dissolved oxygen(19).

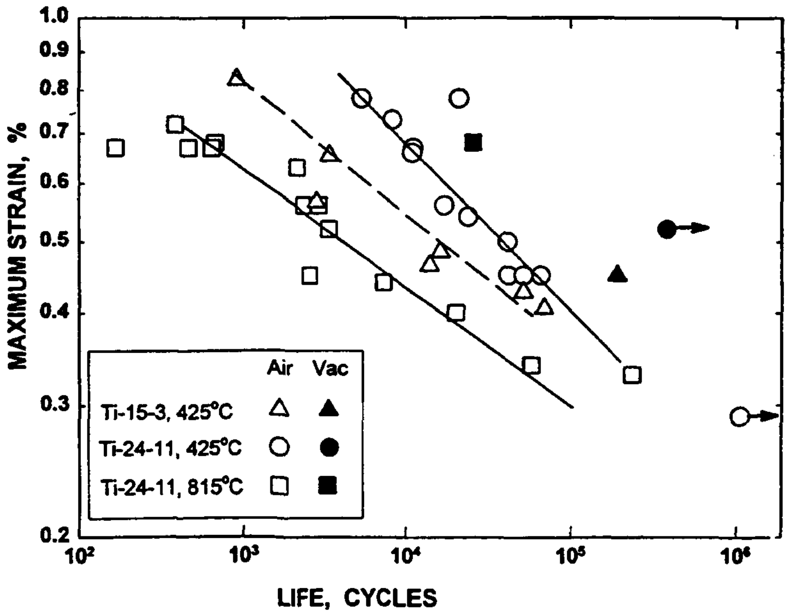


Figure 5. High temperature isothermal fatigue life of Ti matrix composites, showing the degradation in life in air as compared to inert environments, and the relatively minor effect of matrix alloying on life.

3. NiAl Matrix Composites

The turbine and combustor sections of the engine require higher temperature capability , where NiAl is a very attractive candidate(20,21). Specifically for turbine blades and vanes, the competition to NiAl is from superalloy single crystals. NiAl competes well in terms of density, oxidation resistance, and thermal conductivity, and processing appears to be achievable on a commercial level. This indicates that the combination of toughness and creep resistance require the most attention, and approximate goals, as defined in Fig. 6, have been proposed as research and development targets(22). The creep goal has been defined as the stress which is needed to produce a given strain rate at about 1000°C, in density compensated form, must be equivalent to a first generation single crystal superalloy. Although the toughness of superalloys is typically greater than 60 MPa√m, we have chosen 20 MPa√m at room temperature as both a tolerable and achievable goal. These are considered as necessary yet insufficient goals, but it is felt that this emphasis could still provide focus to material development efforts.

Fig. 6 shows that unalloyed NiAl and most polycrystalline alloys have not displayed attractive performance relative to these goals: some very high toughness values have been obtained, but none of those alloys have very good creep strength. Single crystals have been alloyed for very high creep strength which exceed the goal(22,23), and Darolia(20) will be describing this technology in these proceedings. However, these alloys are not very tough, and design changes seem to be the only way to accommodate these low values. This may still be realistic, especially if one can rely on the high ductility of these alloys above their ductile/brittle transition temperature(DBTT). Nevertheless, it is still preferable to have materials that are both tough and creep resistant, and two composite strategies have shown such potential.

NiAl based eutectics have the potential to reach an appropriate balance of properties, and are also included in Fig. 6. NiAl with α-Cr, α-Mo or Cr-Mo solid solutions are two phase eutectics that have been extensively studied(21), and Oliver and co-workers(24,25) have developed a 3 phase eutectic with both α-Cr and the Ta-rich Laves phase coexisting. They have found that the α-Mo and the Cr/Mo eutectic alloys are reasonably tough, and the Ta-rich Laves phase eutectics are very good in creep. The

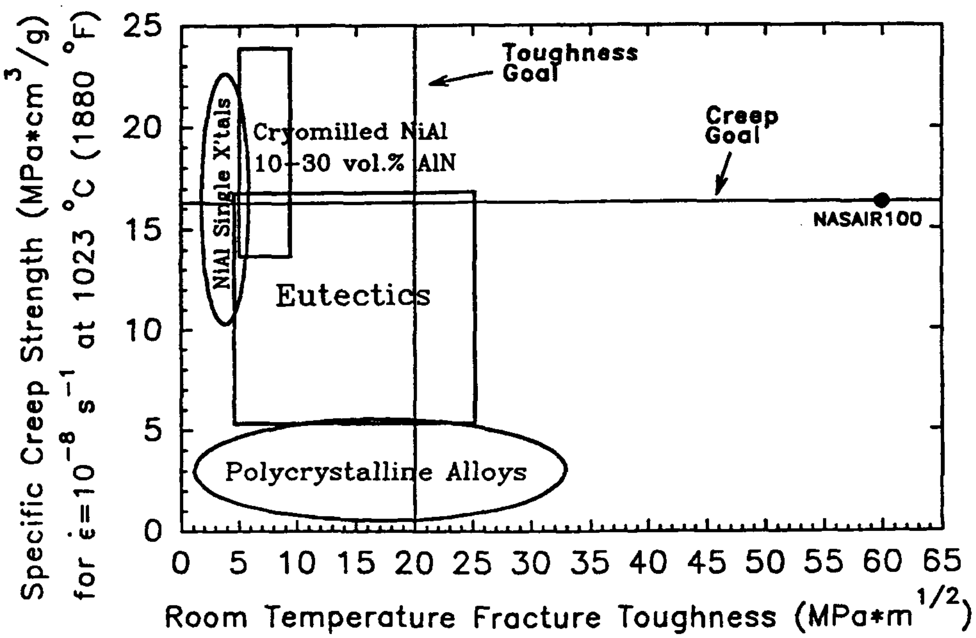


Figure 6. Proposed performance targets for NiAl materials for turbine blade and vane application, along with regimes of capabilities (demonstrated to date) of various material development strategies.

next step of progressing to the 3 phase Laves plus α-Cr showed a substantial gain in toughness at only a small sacrifice in creep strength. This indicates that further alloy development along this line has considerable potential for reaching the balanced creep/toughness goals.

A final alternative is provided by AlN strengthened NiAl, which is produced by high energy milling in liquid N_2, a process termed cryomilling(26). Nitrogen is absorbed into the NiAl powders during cryomilling and forms high volume fractions of AlN in the form of nanometer sized particles. The most desirable microstructure obtained thus far has been obtained by hot extrusion, where the very fine nitride particles are clustered and strung out parallel to the extrusion axis. It is possible to vary the milling conditions to obtain nitride contents ranging from 0 to 30 vol%. It has been found(27) that improvements in creep resistance scaled with the volume fraction of AlN, yet room temperature fracture toughness was not affected by AlN content. This combination of properties is also displayed in Fig. 6, where the cryomilled material showed equivalent creep strength and slightly better toughness compared to the single crystal NiAl alloys. Another advantage of the cryomilled material is its relatively low DBTT. Measurements of fracture toughness as a function of temperature, Fig. 7, indicate that unalloyed NiAl has the lowest DBTT, achieving 20 MPa$\sqrt{m}$ at about 400°C. The cryomilled NiAl+30vol% AlN exhibited a DBTT shifted to about 550°C, which is considerably lower than that seen in many alloys. Usually, alloying elements added for creep strengthening reduce the toughness at room temperature, and raise the DBTT to near 800°C(20,22). A DBTT of 550°C is low enough that the entire turbine blade could be designed to be operated in the ductile regime, thus providing adequate toughness. One strategy for further development would involve the use of the higher nitride contents as a baseline that could tolerate some losses in creep strength in order to improve toughness.

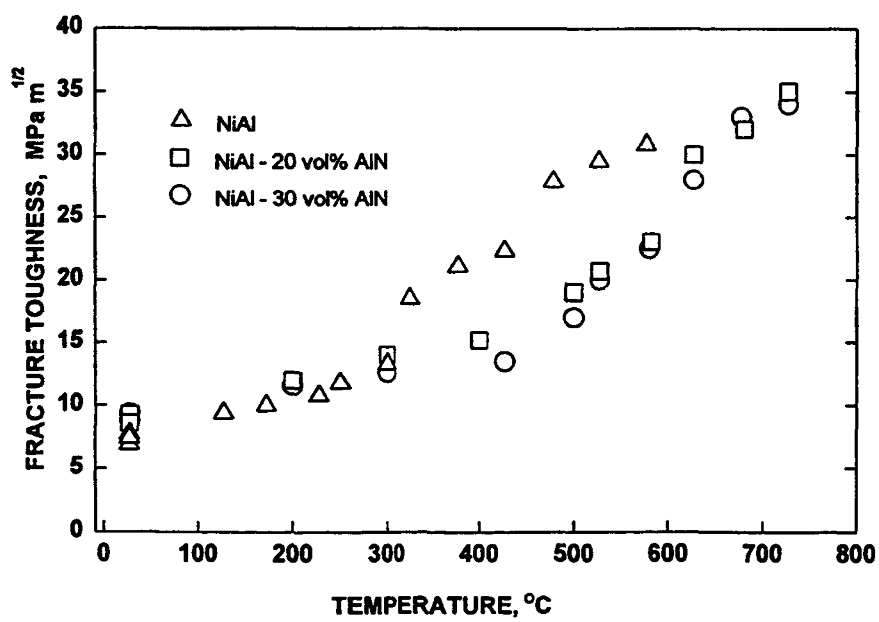


Figure 7. Temperature dependence of fracture toughness for NiAl and NiAl/AlN composites.

4. $MoSi_2$ Matrix Composites

$MoSi_2$ -based composites are a next step over both superalloys and NiAl in terms of temperature capability. Many silicides and other very high temperature intermetallics have been investigated over the past several years, but again the achievement of a balance of properties has been elusive. One promising version, based on a hybrid composite with both Si_3N_4 particles and SiC fibers, has produced one of the better combinations of properties in this general class of material. Hebsur(28) has strengthened $MoSi_2$ by adding about 30 vol% Si_3N_4 in the form of fine particulate. The Si_3N_4 addition has provided three benefits: 1. it strengthens $MoSi_2$ to the extent that the creep strength at temperatures near 1200°C is improved by about five orders of magnitude in steady state creep rate; 2. it lowers the thermal expansion coefficient, so that this two phase particulate composite can be further reinforced with SiC fibers; and 3. it eliminates pesting.

It is generally known that $MoSi_2$ is very good in high temperature oxidation but can disintegrate to powder after only a few hours at 500°C, a phenomenon known as pesting(15,28). Pesting is a complex phenomenon that may be manifested in several different ways. In order to determine if Si_3N_4 additions to $MoSi_2$ have truly eliminated pesting, several different tests were performed(28). First, the traditional meaning of pesting, total disintegration, has been avoided even after 1000 hours at 500°C. However, several workers have achieved this level of pest resistance by merely eliminating porosity, although the fully dense material still exhibits accelerated oxidation due to the formation of MoO_3. The Si_3N_4 /$MoSi_2$ composites, however, exhibited an order of magnitude reduction in oxidation rate at 500°C. Experiments involving pre-cracking followed by oxidation, and applied loads on notched specimens during oxidation, were also run to demonstrate the absence of any MoO_3 formation at a crack tip, which could wedge open the crack and cause premature failure. Finally, $MoSi_2$ /Si_3N_4 composite specimens were subjected to cyclic burner rig tests using jet fuel, again with positive results. This extraordinary improvement in performance has been attributed to the formation of a new protective oxide, Si_2ON_2, that prevents the formation of MoO_3.

This particulate composite with improved environmental resistance and creep strength, has next been used as a matrix for SiC fiber reinforcement in order to improve toughness. Hybrid composites made with 30 vol% Si_3N_4 particulate and 30 vol% SiC fiber have been fabricated and tested in tension, thermal cycling, and chevron notched four-point bending up to 1200°C(29). These composites exhibited ultimate tensile strengths on the order of 700 MPa, and did not exhibit any cracking due to thermal cycling. Furthermore, the SiC fibers provided tremendous crack bridging that allowed for very significant improvements in apparent fracture toughness. Room temperature apparent toughness values of about 35 Mpa√m have been measured, compared to a value of ~ 5 MPa√m for the stand-alone nitride particulate strengthened matrix. In summary, a composite strategy has again been used to achieve a better balance of properties.

5. Summary

Several composites have been developed that show promise for applications throughout a jet engine, as summarized in Table I. Titanium matrix composites reinforced with continuous SiC fibers are attractive in the compressor and the fan because of their excellent stiffness and strength, especially on a density-compensated basis. These composites are competing with monolithic Ti and Ni alloys, in addition to TiAl in the hotter parts and polymer composites in the colder parts. These composites are limited to about 600°C primarily because of their environmental resistance and their transverse properties, but still look quite attractive for several applications. About the only serious barrier for these composites is cost, which is high primarily because of manufacturing costs and fiber costs. NiAl looks promising at temperatures up to about 1100°C, which is appropriate for the turbine and the combustor, and can compete with superalloys because of advantages in thermal conductivity, environmental resistance, and specific strength. Its main barrier is toughness, and both directionally solidified eutectics and AlN particle-strengthened NiAl show promise in achieving a balance of both strength and toughness. Finally, less mature, but capable of still higher temperatures, is $MoSi_2$, particularly a hybrid composite with both Si_3N_4 particles and SiC fibers. This composite must compete against superalloys, other silicides, and ceramic composites. This composite has excellent environmental resistance compared to all other silicides, has better toughness, thermal conductivity, and processability compared to ceramics, and has much higher strength at high temperatures compared to superalloys. In order to outdo the competition, however, it is still necessary to improve creep strength, especially in light of ceramics, and toughness and cost compared to superalloys.

TABLE I
Summary of Capabilities and Applications of High Temperature Composites

COMPOSITE SYSTEM	APPLICATION/ TEMPERATURE LIMIT	BENEFITS	BARRIERS
Titanium	Compressor Fan T≈ 600°C	- Stiffness - Strength	- Environmental embrittlement - Transverse properties - Cost
NiAl	Turbine Combustor T≈ 1100°C	- Conductivity - Environmental - Strength	- Toughness
$MoSi_2$	Turbine Combustor T≈ 1250°C	- Environmental - K_{IC}, conductivity, & processing *vs* ceramics - Temp/strength capability *vs* superalloys	- Creep *vs* ceramics - Toughness & cost *vs* superalloys

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13. ABSTRACT (*Maximum 200 words*)

The purpose of this paper is to review the current state of development of new composite materials for advanced aircraft engines. The advantages and disadvantages of Ti-base, NiAl-base, and $MoSi_2$-base composites as replacements for today's Ni-base superalloys are discussed from the standpoint of key technical issues, current status, and future directions. Results describing progress in both improved understanding of the mechanisms of deformation and fracture, and improved material performance will be covered.

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